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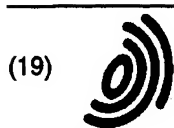
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(54) **Process for the manufacture of wood composition boards, prepress sealer compositions and use thereof**

Verfahren zur Herstellung von Spanplatten und einem Bindemittel zur Behandlung der Holzspäne vor dem Pressen

Procédé de fabrication de panneaux à base de bois et d'une composition liante utilisée pour le traitement du matelas avant pressage

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## Description

This invention is concerned with a method for preparing wood composition boards, such as hardboard, fabricated consolidating fibers or chips under heat and pressure to form an integral board material. The method incorporates applying an aqueous polymeric composition, also known as a prepress sealer, to the surface of a fibrous mat prior to subjecting the mat to a high temperature and pressure press treatment.

Wood composition board such as, for example, hardboard is manufactured by the reconsolidation of defiberated wood chips under heat (up to about 450 F. (232°C)) and pressure (up to about 1000 p.s.i. (6.895 x 10<sup>6</sup> Pa)). The fibrous particles are first formed into a unitary mat (either by a dry process, wet process, or wet-dry process) which is then consolidated into a solid board by applying heat and pressure. Prior to the consolidation, compositions containing aqueous polymeric dispersions, aminoplast resins, phenol-formaldehyde resins, waxes and the like may be applied to the mat to increase strength, improve surface integrity, and enhance water resistance. These compositions are referred to as "prepress sealers". However, the production of wood composition boards still suffers technical problems such as press plate sticking, loss of press plate definition, lack of board surface toughness and uniformity, excessive water absorptivity, and discoloration. Further, repetitive consolidation cycles may cause deposits of prepress sealer and fibers on the press plates. These deposits may require frequent plate cleaning and lead to lower production rates as a result of increased downtime.

GB-A-1,484,053 discloses impregnating a paper carrier web with an aqueous solution of an aminoplast resin, and drying the impregnated web in such a way that it contains 40 to 60 % by weight of aminoplast resin precondensate, relative to the weight of the paper, and coating the dried impregnated web with an aqueous dispersion comprising a curable resin composition which is a mixture of A) a hard and brittle copolymer which has been obtained by copolymerization of certain monomers, and B) a rubbery-elastic copolymer which has a glass transition temperature not higher than +10 C., and has been obtained by the copolymerization of certain monomers, including 0 to 2% by weight of a crosslinking monomer with at least two reactive non-conjugated double bonds in the molecule. Further disclosed is coating the impregnated paper web and pressing the resulting film onto a pressboard sheet. This reference does not disclose the application of a polymer dispersion/aminoplast mixture to a fibrous mat prior to compaction.

US-A-4,201,802 discloses a process for manufacturing prefinished hardboard which includes the use of an aqueous pre-press sealer containing a polyvinyl alcohol polymer together with emulsified fatty acids or esters, a volatile amine, and aluminum, zinc, or calcium stearates as well as driers or catalysts if desired. The polyvinyl alcohol is disclosed to be preferably "a homopolymer of vinyl alcohol monomer copolymerized to provide a polymer although a copolymer can be used containing very minor amounts up to about 5% of other ethylenically unsaturated monomers containing carbon-to-carbon double bond unsaturation." In a further disclosure the polyvinyl alcohol is described as partially hydrolyzed, preferably 85 to 90% hydrolyzed. This reference does not teach or suggest the importance of the gel content of the dispersed polymer.

US-A-4,238,438 discloses a pre-press hardboard treatment comprising applying to the surface fibers of a fibrous mat from 8 to 30 grams per square foot (0.093m<sup>2</sup>) of a coating of a surface consolidating agent comprising from 50 to 95% water, from 4 to 25% of a hydroxy- radical-containing compound having a boiling point between about 212 F (100°C) and 600 F (315°C), which is selected from the group of alcohols and their esters, and from 1 to 25% polyvinyl acetate, wherein the percentage of coating ingredients are based on the weight of the coating. The polyvinyl acetate latex emulsions disclosed are commercial materials. The reference is silent with regard to specific compositions or gel content of the polyvinyl acetate latex.

US-A-4,336,174 and US-A-4,374,899 disclose a pre-press hardboard treating composition and an improved hardboard sheet produced by the coating of the wet felt mats or laps or sheets, before consolidation and drying, with an aqueous sealer composition that consists essentially of (a) blend of water-soluble melamine-formaldehyde copolymer, (b) a styrene-acrylic copolymer emulsion, (c) a small amount of an amine capable of adjusting the pH of the resulting emulsion, and (d) a crosslinking acid catalyst capable of cross-linking the composition. Disclosed are the use of commercial vinyl acetate and styrene/acrylic copolymer emulsions. The reference is silent regarding the compositions of the copolymer emulsions used or their gel content.

US-A-4,517,228 discloses a conventional process for preparing composition board products which employs a unique step of applying to a composition mat of a density of less than 60 lbs. per cubic foot (961 kg m<sup>-3</sup>) prior to the application of heat and pressure, a coating composition comprising an hydroxyl or carboxylic acid-containing acrylic vehicle, a high melting wax, a melamine-based crosslinker for the vehicle, and a platlet form of talc. The hydroxyl or carboxylic acid acrylic vehicle is not disclosed to contain any gel content.

US-A-4,517,240 discloses a one-step process for simultaneously compressing and tempering a fiberboard panel, which includes the step of applying to the panel prior to compression thereof an effective amount of an aqueous treating composition which contains about 3-20% by weight of an acrylic or vinyl acetate emulsion polymer or solution polymer and about 0.05 to 3.0% by weight of a fluid, water-soluble organosilicone copolymer of dimethylpolysiloxane and a polyoxyalkylene ether wherein the alkylene moiety is ethylene or propylene or mixtures thereof, characterized in that

the treating composition acts both as a platen release agent during the compression and as a tempering agent for imparting water resistance. Acrylic polymers included alkyl methacrylate/alkyl acrylate/acrylic or methacrylic acid and styrene/alkyl acrylate/N-alkylol acrylamide/ acrylic or methacrylic acid copolymers, for example. The polymers were not disclosed to include any gel content.

US-A-4,075,141 discloses coating compositions which contain crosslinked polymeric microparticles and a carboxylic acid amide interpolymers which may be combined with an aminoplast resin crosslinking agent. The compositions are disclosed to be useful for application to substrates such as paper, metal, wood, paperboard, plastic, foam, extruded rubber, and the like. Neither specific gel content nor use on wood composition board are disclosed.

US 4,062,823 discloses compositions incorporating a mixture of at least two copolymers of acrylic monomers, a crosslinked latex, an amine, an amino resin crosslinking agent, and water or water and an organic solvent. The use of these compositions as water-based paints is disclosed. Neither specific gel content of the crosslinked latex nor use on wood composition board are disclosed.

None of the references teach the use of a prepress sealer composition which contains an aminoplast resin and an aqueous dispersion of acrylic particles with a gel content greater than about 70% for application to a fibrous mat prior to the consolidation of a wood composition board.

An object of this invention is to provide a prepress sealer composition which provides improved press release during the process of manufacturing wood composition boards.

It is a further object of this invention to provide an improved hardboard with superior water resistance and surface toughness by the use of a prepress sealer composition.

In accordance with the present invention a prepress sealer composition is provided. When the prepress sealer is used in the process of making wood composition board, the process is improved as a result of more facile release of the formed composition board from the press plates. When the prepress sealer is used, the water resistance and surface toughness properties of the formed board are also improved.

In another aspect, this invention is directed to a process for manufacturing wood composition boards under heat and pressure comprising providing a fibrous mat, treating the surface of said mat, hot pressing said mat at elevated temperature and pressure into a solid composition board product, and releasing said composition board from said press characterised in that said process comprises treating the surface of said mat with a composition comprising a crosslinking resin which comprises an aminoplast resin, multi-functional hydroxyalkyl amide or multi-functional N-alkylol amide, and an aqueous dispersion of acrylic polymer particles; wherein said particles have a THF gel content as determined by the method defined hereinabove of 67% or more, preferably greater than 70%, and an acrylic content of at least 20% by weight based on the total weight of polymer.

As used herein, "wood composition board" includes the various hardboards, fiber boards, particle boards, wafer boards, and strand boards, such as, for example, wet processed hardboards, dry processed hardboard, wet/dry processed hard boards, medium density fiber board, oriented strand board, and mende boards.

The prepress sealer incorporates a crosslinking resin, that is, a species adapted to react with functionality incorporated in or associated with the acrylic polymer particles under the conditions attained during the manufacture of the wood composition board. One class of crosslinking resins suitable for use in this invention are aminoplast resins such as, for example a melamine-formaldehyde crosslinking agent, a urea-formaldehyde crosslinking agent, or mixtures thereof. Other crosslinking resins which may be used in the practice are multi-functional resins such as, for example, multi-functional hydroxyalkyl amides and multi-functional N-alkylol amides.

The prepress sealer incorporates an aqueous dispersion of acrylic polymer particles wherein the acrylic polymer particles have a THF gel content of greater than 70 %. The aqueous dispersion of acrylic polymer particles may be prepared by any aqueous polymerization technique known in the art such as, for example, suspension polymerization and emulsion polymerization. Emulsion polymerization is preferred. The acrylic polymer particles have an acrylic content of at least 20% by weight of the total weight of the polymer. Preferred is an acrylic content of at least about 40% by weight of the total weight of the polymer. The acrylic polymer particles are prepared by the polymerization of ethylenically unsaturated monomers. The acrylic content results from the polymerization of acrylate or methacrylate esters, amides, acids, and the like, such as, for example, methyl acrylate, ethyl acrylate, butyl acrylate, 2-ethylhexyl acrylate, decyl acrylate, methyl methacrylate, ethyl methacrylate, butyl methacrylate, i-butyl methacrylate, i-bornyl methacrylate, acrylamide, acrylonitrile, hydroxyethyl methacrylate, hydroxypropyl methacrylate, methylol (meth)acrylamide, methylolated ureidoethyl methacrylate, N,N-dimethylaminoethyl methacrylate, acrylic acid, methacrylic acid, and acryloxypropionic acid. The non-acrylic portion of the acrylic polymer particles results from the polymerization of styrenes and other ethylenically unsaturated monomer such as, for example, styrene, substituted styrenes, butadiene, vinyl acetate, vinyl versatate, crotonic acid esters, maleic acid, fumaric acid, and itaconic acid.

The gel content of the acrylic polymer particles may relate to crosslinking of the polymer molecules within the particles. Such crosslinking may occur during or subsequent to the preparation of the particles, or by a combination thereof. Crosslinking may be effected by the incorporation of multi-ethylenically unsaturated monomers into the polymerization. The multi-ethylenically unsaturated monomers may be acrylic such as, for example, 1,4-butyleneglycol

dimethacrylate, trimethylolpropane triacrylate, and allyl methacrylate or non-acrylic such as, for example, diallyl phthalate and divinyl benzene. Gel content is preferably effected by the incorporation of allyl methacrylate into the acrylic polymer particles at a preferred level of about 0.2% to about 5% by weight based on the weight of the polymer particles. More preferred is the incorporation of allyl methacrylate into the acrylic polymer particles at a level of about 0.3% to about 2.0% by weight based on the weight of the polymer particles.

The glass transition temperature (T<sub>g</sub>) of the acrylic polymer particles may be in the range of from 15 C to 100 C., as determined by differential scanning calorimetry. Preferred is a T<sub>g</sub> of from 20 C to 50 C.. The acrylic polymer particles may include blends of polymers; incorporating polymer(s) with a T<sub>g</sub> greater than 50 C. in blends is useful in increasing the surface hardness of the resulting board.

It is required that the acrylic polymer particles have a THF gel content greater than 70%. The THF gel content as defined herein was determined by preparing a 2% mixture of polymer particle solids in 1 ounce (29.6 ml) of tetrahydrofuran. The exact solids of the mixture was determined by drying the solution at 150°C. for a period of 30 minutes. The 2% mixture was shaken overnight and transferred to a tared centrifuge tube. The tube was spun down on an ultracentrifuge at 50,000 rpm for 2.5 hrs. The supernatant solution was decanted off, most of the THF was allowed to evaporate at room temperature, and then dried down at 150°C. for 30 minutes. Duplicate runs were carried out. The THF gel fraction was the proportion of polymer which was not dissolved in the supernatant solution.

The prepress sealer of this invention may also be compounded with waxes, pigments, fillers, reinforcing agents, flow control agents, release agents such as, for example, calcium stearate, antifoaming agents, and the like. The prepress sealer may be applied to the surface of the mat by conventional techniques such as, for example, spraying.

The following examples are intended to illustrate the improvement in the process for manufacturing wood composition boards to which this invention is directed. They are not intended to limit the invention as other applications of the invention will be obvious to those of ordinary skill in the art.

#### EXAMPLE 1. Preparation of aqueous copolymer dispersion

Preparation of Sample 1. To a one gallon stirred reactor containing 1006.26 grams(g.) of deionized water (DI water), 4.0 g. of anionic surfactant (58% active), and 27.4 g. of itaconic acid which had been heated to 85.2 C. was added 128.3 g. of a preform emulsion and a solution of 4.6 g. ammonium persulfate in 29.8 g. DI water. The temperature dropped to 80.5 C. and then an exotherm to 83.2 C was observed, along with a blue color and heavy refluxing. Fifteen minutes after the addition of the preform the temperature was at 81 C. and the addition of the monomer emulsion (ME#1) and a solution of 2.7 g. ammonium persulfate in 98.7 g. DI water were begun. The addition of ME#1 was completed in 142 minutes and a rinse of 22.3 g. DI water was added. The addition of the ammonium persulfate solution was completed 38 minutes later with the temperature at 81 C. and cooling was initiated. Nine minutes later, with the temperature at 74.2 C., 37.2 g. of DI water was added and cooling was continued. Six minutes later, with the temperature at 60 C., three solutions were added: 0.9 g. t-butyl hydroperoxide and 0.15 g. anionic surfactant (58 % active) in 9.3 g. DI water; 0.5 g. sodium sulfoxylate formaldehyde in 14.0 g. DI water; and 4.65 g. of a 0.15% aqueous solution of ferrous sulfate heptahydrate. Fifteen minutes later with the temperature at 55 C., solutions identical to the first two of the three abovementioned solutions were added. Twenty-two minutes later, with the temperature at 50 C., 53.9 g. DI water, 36.7 g. dimethylaminoethanol, and 15.35 g. of anionic surfactant were added. The final product had a solids content of 46.8%, a pH=8.75, a viscosity of 435 centipoises, a particle size of 121 nanometers, and a THF gel content of 67.

Table 1.1

Monomer Emulsion ME#1	
Butyl acrylate	780.3 g.
Methyl methacrylate	477.1 g.
Styrene	459.4 g.
Hydroxyethyl Methacrylate	92.1 g.
Allyl methacrylate	4.6 g.
Anionic surfactant(58% active) (ALIPAL CO436)	27.5 g.
DI water	688.2 g.

#### EXAMPLE 2. Preparation of aqueous copolymer dispersions

Preparation of Samples 2-4 and Comparative Sample A. The samples were prepared according to the method of

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Example 1 with the exception that a different monomer emulsion (ME#1) was used in each case as indicated in Table 2.1 and that the polymerization of Sample 2 was carried out in a 5 gallon reactor with a five-fold increase in the quantities of all ingredients. The resulting dispersions exhibited the properties tabulated in Table 2.2.

Table 2.1

Monomer Emulsions ME#1 (All quantities are in grams)				
Sample	2	3	4	Comparative A
Butyl acrylate	3901.4	780.3	780.3	780.3
Methyl methacrylate	2385.5	477.1	477.1	477.1
Styrene	2297.1	459.4	459.4	459.4
Hydroxyethyl methacrylate	460.4	92.1	92.1	92.1
Allyl Methacrylate	45.9	18.4	36.7	-
Anionic surfactant (ALIPAL CO436)	137.7	27.5	27.5	27.5
DI water	3441.0	688.2	688.2	688.2

Table 2.2

Properties of the aqueous dispersions of Example 2.				
Sample	2	3	4	Comparative A
Wt. % solids	46.8	47.0	47.1	46.0
pH	8.6	8.8	8.7	8.5
Viscosity (cps.x 10 <sup>-3</sup> PaS)	325	450	495	215
Particle size(nm.)	124	118	113	125
THF gel content(%)	85	92	92	3

## EXAMPLE 3. Preparation of all-acrylic aqueous dispersions

Preparation of Samples 5-8 and Comparative Sample B. To a one gallon stirred reactor containing 1113 grams(g.) of deionized water (DI water), 4.1 g. of anionic surfactant (58% active), and 28.3 g. of itaconic acid which had been heated to 85.0 C. was added 130 g. of a preform emulsion and a solution of 4.7 g. ammonium persulfate in 30 g. DI water. The temperature dropped to 80.5 C. and then an exotherm to 85.0 C was observed, along with a blue color and heavy refluxing. Fifteen minutes after the addition of the preform the temperature was at 81 C. and the addition of the monomer emulsion (ME#1) and a solution of 2.8 g. ammonium persulfate in 106.2 g. DI water were begun. The addition of ME#1 was completed in 171 minutes and a rinse of 40 g. DI water was added. The addition of the ammonium persulfate solution was completed five minutes later: the temperature was 81 C.. Fifteen minutes later, with the temperature at 81 C., 40 g. of DI water was added and cooling was initiated. Six minutes later, with the temperature at 54.5 C., three solutions were added: 0.95 g. t-butyl hydroperoxide in 10 g. DI water; 0.5 g. sodium sulfoxylate formaldehyde in 15 g. DI water; and 5 g. of a 0.15% aqueous solution of ferrous sulfate heptahydrate. Fifteen minutes later with the temperature at 50.5 C., solutions identical to the first two of the three abovementioned solutions were added. Twenty-five minutes later the temperature was 44.5 C. and 70.7 g. DI water, 37.7 g. dimethylaminoethanol, and 15.7 g. anionic surfactant were added.

Table 3.1

Monomer Emulsions (ME#1) used in Example 3.					
Sample	5	6	7	8	Comparative B
Butyl Acrylate	802	802	802	802	802
Methyl methacrylate	962	962	962	962	962
Hydroxyethyl methacrylate	94.3	94.3	94.3	94.3	94.3
Allyl methacrylate	4.7	9.4	18.9	37.7	-
Anionic surfactant (ALIPAL CO436)	28.5	28.5	28.5	28.5	28.5
DI water	704	704	704	704	704

Table 3.2

Properties of aqueous dispersions of Example 3.					
Sample	5	6	7	8	Comparative B
Wt. % solids	45.4	45.5	45.9	45.7	46.0
pH	8.75	8.8	8.8	8.75	8.6
Viscosity(cps. x 10 <sup>-3</sup> PaS)	210	150	115	160	-
Particle size(nm.)	165	130	110	135	127
THF gel content(%)	89	91	94	94	4

## EXAMPLE 4. Preparation of high hydroxyl content aqueous dispersions

Preparation of Samples 9-10 and Comparative Sample C. To a one gallon stirred reactor containing 1300 grams (g.) of deionized water (DI water), 4.0 g. of antioxidant(MAROXOL 20) and 30 g. of itaconic acid which had been heated to 85 C. was added 150 g. of a preform emulsion(45.5% solids) and a solution of 5 g. ammonium persulfate in 50 g. DI water. The temperature dropped to 81 C. and then an exotherm to 83 C was observed, along with heavy refluxing. Five minutes after the addition of the preform the temperature was at 83 C. and the addition of the monomer emulsion (ME#1) and a solution of 3 g. ammonium persulfate in 150 g. DI water were begun. The addition of ME#1 and the ammonium persulfate solution were completed in 180 minutes and a rinse of 50 g. DI water was added. Thirty minutes later the temperature was at 79 C., 50 g. DI water was added, and cooling was initiated. Thirty-five minutes later with the temperature at 55 C., three solutions were added: 1.0 g. t-butyl hydroperoxide in 5.0 g. DI water; 0.6 g. sodium sulfoxylate formaldehyde in 15.0 g. DI water; and 7 g. of a 0.15% aqueous solution of ferrous sulfate heptahydrate. Ten minutes later with the temperature at 53 C., solutions identical to the first two of the three abovementioned solutions were added. Twenty-six minutes later, with the temperature at 45 C., 50 g. DI water, 30 g. dimethylaminoethanol, and 15 g. of anionic surfactant were added.

Table 4.1

Monomer Emulsions(ME#1) for Example 4			
Sample	9	10	Comparative C
Butyl acrylate	850	850	850
Methyl methacrylate	210	180	220
Styrene	500	500	500
Hydroxyethyl methacrylate	400	400	400
Allyl methacrylate	10	40	-
Anionic Surfactant (TRITON XN-45S; 60% active)	33	33	33
DI water	1300	1300	1300

Table 4.2

Properties of aqueous dispersions of Example 4			
Sample	9	10	Comparative C
Wt. % solids	46.1	46.1	46.1
pH	7.4	7.5	7.5
Viscosity(cps.x 10 <sup>-3</sup> PaS)	403	259	647
Particle size(nm.)	256	282	284
THF gel content(%)	90	95	32

## EXAMPLE 5. Preparation of intermediate hydroxyl level aqueous dispersions

Preparation of Samples 11-14. To a one gallon stirred reactor containing 700 grams(g.) of deionized water (DI water), 4.0 g. of antioxidant (MAROXOL 20; 0.5%), and 30 g. of itaconic acid which had been heated to 85 C. was

added 150 g. of a preform emulsion and a solution of 5 g. ammonium persulfate in 50 g. DI water. The temperature dropped to 75 C.. The reaction mixture was heated to 82 C. Four minutes after the addition of the preform the temperature was at 82 C. and the addition of the monomer emulsion (ME#1) and a solution of 3 g. ammonium persulfate in 150 g. DI water were begun. The addition of ME#1 and the ammonium persulfate solution was completed in 180 minutes and a rinse of 50 g. DI water was added. Thirty minutes later cooling was initiated. Then, 50 g. of DI water was added and cooling was continued. Twenty minutes later, with the temperature at 55 C., three solutions were added: 1.0 g. t-butyl hydroperoxide (70% active) in 10 g. DI water; 0.6 g. sodium sulfoxylate formaldehyde in 15 g. DI water; and 7 g. of a 0.15% aqueous solution of ferrous sulfate heptahydrate. Ten minutes later with the temperature at 55 C., solutions identical to the first two of the three abovementioned solutions were added. Twenty minutes later, with the temperature at 45 C., 50 g. DI water, 30 g. dimethylaminoethanol, and 15 g. of anionic surfactant (30% active) were added.

Table 5.1

Monomer Emulsions(ME#1) for Example 5				
Sample	11	12	13	14
Butyl acrylate	850	850	850	850
Methyl methacrylate	510	480	410	380
Styrene	500	500	500	500
Hydroxyethyl methacrylate	100	100	200	200
Allyl methacrylate	10	40	10	40
Anionic Surfactant (TRITON XN-45S; 60% active)	33	33	33	33
DI water	600	600	600	600

Table 5.2

Properties of aqueous dispersions of Example 5				
Sample	11	12	13	14
Wt. % solids	53.5	53.5	53.5	53.5
pH	7.7	7.6	7.8	7.4
Viscosity(cps.x10 <sup>-3</sup> PaS)	165	202	1432	740
Particle size(nm.)	293	306	271	283
THF gel content(%)	87	93	88	91

## EXAMPLE 6. Evaluation of press release

To 100 g. of aqueous dispersion solids was added 10 g. of aminoplast resin(CYMEL 303; 100% active) and 10 g. of Calcium Stearate(50% solids active) with stirring. The mixture ("prepress sealer") was diluted to 20% total solids with DI water. The prepress sealer was sprayed uniformly at an add-on of 10 grams per square foot onto a dry process fiber mat containing resin binder, wax, and moisture. The fiber mat was placed in a press with a 450 F. (232°C) platten temperature and subjected to 380 p.s.i. (2.62 x 10<sup>6</sup>Pa) pressure for 1 minute. The resultant board product had a density of about 1.05 and was about 1/8 inches (0.32 cm) thick. The press releasing performance was immediately after the completion of the press cycle, i.e., when the press pressure reached zero, using the following scale:

Rating	Observation
10	Board releases and falls immediately
9	Board sticks and then falls
8	Board sticks but is easily loosened
7	Board sticks but requires moderate force to loosen
6	Board sticks and must be pried off
3	Very firmly stuck
1	Very stuck and requires chemical soak or sand-blasting to clean the plates



Table 6.1

Evaluation of aqueous dispersions of Examples 1 and 2					
Sample	1	2	3	4	Comparative A
Press Release Rating	9	9.5	10	10	7.5

The samples of this invention demonstrate superior press release.

Table 6.2

Evaluation of the aqueous dispersions of Example 3					
Sample	5	6	7	8	Comparative B
Press Release Rating	8.5	9.5	10	10	7

The all-acrylic samples of this invention demonstrate superior press release.

Table 6.3 Evaluation of the aqueous dispersions of Examples 4 and 5

Sample	9	10	11	12	13	14	Comparative C
Press Release Rating	10	10	10	10	10	10	8

The high hydroxyl functionality, which may lead to a higher level of crosslinking with the aminoplast resin (Comparative C), does not achieve the superior press release of the samples of this invention.

#### EXAMPLE 7. Evaluation of water uptake of wood composition boards

The water uptake of wood composition boards was measured by the Cobb test. A Cobb unit is defined as the amount of distilled water weight increase in grams per 100 square inches of surface area per 24 hour period.

Boards for measurement were cut into 5 inch by 5 inch squares. An Aluminum ring of 4 inch inside diameter, wall thickness of 3/8 inch and height of one inch was affixed to the test boards with beads of butyl caulk(which had been determined not to contribute to weight change). The boards with rings and control boards without rings were equilibrated for seven days at 65-75 C. and 50-60 % R.H. The boards with rings and the control boards were weighed. The ring was filled with approximately 1/2 inch of distilled water. After 24 hours the water was poured out, the ring surface was blotted dry with a soft paper towel, and the board with ring was weighed. The control board was then weighed; if the weight of the control board had changed by more than 0.20 g., the entire test was abandoned. The weight change for the board with ring was multiplied by 7.96 to yield the Cobb unit value.

Table 7.1 Evaluation of Cobb water pick up

Samples 9-14 and Comparative C were formulated into prepress sealers according to the method of Example 6 and evaluated for Cobb water pick-up.

Sample	9	10	11	12	13	14	Comp.C	Water
Cobb Units	21.79	22.65	17.71	23.25	15.20	22.65	17.40	34.04

All of the prepress sealers have substantially lower water pickup than when water is used as the prepress sealer, that is, all function to render the formed board more water resistant.

#### EXAMPLE 8. Evaluation of surface integrity

The surface integrity of wood composition boards to which a prepress sealer had been applied was evaluated by a tape adhesion test. A board sample approximately 4 inches by 6 inches (10.2cm x 15.2 cm) was selected. A strip of tape (3M tape #610 was used) was placed on the surface of the board and pressed on the surface by rubbing it with the tape roll. One end of the tape was pulled off with a quick steady motion with the pulled end of the tape at a 90 degree angle to the tape adhering to the board. The % of fiber pull was evaluated visually.

Table 8.1

Evaluation of the surface integrity of boards treated with prepress sealer							
Sample	9	10	Comp.C	11	12	13	14
% Fiber Pull	0	0	20	0	5	0	2

The samples of this invention display a higher surface integrity than the comparative sample as evidenced by lower levels of fiber pull in the surface integrity test.

#### EXAMPLE 9. Prepress sealers based on aqueous copolymer dispersions incorporating different acids.

Preparation of Samples 15 and 16. To a one gallon stirred reactor was containing 1063 grams(g.) of deionized water (DI water) and 4.0 g. of anionic surfactant (60% active) which had been heated to 85 C. was added 82 g. of a preform emulsion, 8 g. DI water, and a solution of 4 g. ammonium persulfate in 40 g. DI water. The temperature dropped to 81.5 C. and then an exotherm to 86.5 C was observed. Twenty-four minutes after the addition of the preform the temperature was at 82.5 C. and the addition of the monomer emulsion (ME#1) and a solution of 2.4 g. ammonium persulfate in 120 g. DI water were begun. The addition of ME#1 and the persulfate solution were complete in 180 minutes and 10 g. DI water was added. Thirty minutes later with the temperature at 84 C., cooling was initiated. Nine minutes later, with the temperature at 74.2 C., 37.2 g. of DI water was added and cooling was continued. Thirty-nine minutes later, with the temperature at 54.5 C., three solutions were added: 0.8 g. t-butyl hydroperoxide in 4 g. DI water; 0.48 g. sodium sulfoxylate formaldehyde in 12 g. DI water; and 5.6 g. of a 0.15% aqueous solution of ferrous sulfate heptahydrate. Seventeen minutes later with the temperature at 51.5 C., solutions identical to the first two of the three abovementioned solutions were added. Twenty minutes later, with the temperature at 44 C., 40 g. DI water, 28 g. dimethylaminoethanol, and 12 g. of anionic surfactant (30% active) were added.

Table 9.1

Monomer Emulsions(ME#1) for Example 9		
Sample	15	16
Butyl acrylate	680	680
Methyl methacrylate	384	384
Styrene	400	400
Hydroxyethyl methacrylate	80	80
Allyl methacrylate	32	32
Methacrylic acid	24	-
Acrylic acid	-	24
Anionic Surfactant (TRITON XN-45S; 60% active)	22.7	22.7
DI water	420	420

Table 9.2

Properties of aqueous dispersions of Example 9		
Sample	15	16
Wt. % solids	45.8	45.9
pH	9.3	9.0
Viscosity(cps.(x10 <sup>-3</sup> PaS))	413	388
Particle size(nm.)	111	115
THF gel content(%)	90	92

Table 9.3

Properties of prepress sealers of Example 9			
Samples 15, 16, and 4 were formulated into prepress sealers and evaluated for press release as described in Example 6.			
Sample	15	16	4
Copolymerized acid	Methacrylic	Acrylic	Itaconic
THF gel content	90	92	92
Press release	10	10	10

The nature of the copolymerized acid in the samples of this invention does not affect their adhesion to the press plates sufficiently to reduce their excellent press release.

#### EXAMPLE 10. Prepress sealers based on other crosslinking agents

Preparation of Samples 17 and 18. To a one gallon (3.78l) stirred reactor containing 1000 grams(g.) of deionized water (DI water) and 0.6 g. of anionic surfactant (60% active) been heated to 84.5 C. was added 100 g. of a preform emulsion and a solution of 4.25 g. of ammonium persulfate in 25 g. DI water. The temperature dropped to 78 C. and then an exotherm to 84 C was observed. Seventeen minutes after the addition of the preform the temperature was at 83 C. and the addition of the monomer emulsion (ME#1) and a solution of 2.7 g. ammonium persulfate in 100 g. DI water were begun. The addition of ME#1 and the persulfate solution was completed in 180 minutes and 100 g. DI water was added. Thirty minutes later with the temperature at 84 C., cooling was initiated. Ten minutes later, with the temperature at 60 C., 100 g. of DI water was added and cooling was continued. Twelve minutes later, with the temperature at 50 C., three solutions were added: 1.0 g. t-butyl hydroperoxide in 10 g. DI water; 0.5 g. isoascorbic acid in 15 g. DI water; and 5 g. of a 0.15% aqueous solution of ferrous sulfate heptahydrate. Twenty minutes later with the temperature at 42 C., solutions identical to the first two of the three abovementioned solutions were added. Twenty minutes later, with the temperature at 44 C., 25 g. of aqueous ammonia in 100 g. DI water was added.

Table 10.1

Monomer Emulsions(ME#1) for Example 10		
Sample	17	18
Butyl acrylate	786.6	786.6
Methyl methacrylate	498.6	462.6
Styrene	450	450
Allyl methacrylate	-	36
Acrylic acid	64.8	64.8
Anionic Surfactant	29.4	29.4
(TRITON XN-45S; 60% active)		
DI water	650	650

Table 10.2

Properties of aqueous dispersions of Example 10		
Sample	17	18
Wt. % solids	45.1	45.2
pH	7.4	7.8
Viscosity(cps.x10 <sup>-3</sup> PaS)	306	200
Particle size(nm.)	159	165
HF gel content(%)	17	95

Table 10.3

Press release using a hydroxyalkylamide crosslinking resin				
Samples 17 and 18 were formulated into prepress sealers and evaluated for press release as described in Example 6, with the exception that bis(N,N-di(b-hydroxyethyl))adipamide ("HEA") was substituted for the CYMEL 303 as the crosslinking resin. The preparation of the adipamide is described in US-A-4,076,917.				
Sample	17	17	18	18
Crosslinker	None	HEA	None	HEA
Press release	1	2	7	10

Sample 17 which has a gel content outside the scope of this invention exhibits very poor press release with or without crosslinking resin. Sample 18 of this invention exhibits poor press release without crosslinking resin, but excellent press release when formulated into a prepress sealer with a crosslinking resin.

### Claims

1. A process for manufacturing wood composition boards under heat and pressure comprising providing a fibrous mat, treating the surface of said mat, hot pressing said mat at elevated temperature and pressure into a solid composition board product, and releasing said composition board from said press, characterised in that said process comprises treating the surface of said mat with a composition comprising a crosslinking resin which comprises an aminoplast resin, multi-functional hydroxyalkyl amide or multi-functional N-alkylol amide, and an aqueous dispersion of acrylic polymer particles; wherein said particles have a THF gel content as determined by the method defined hereinabove of 67% or more, preferably greater than 70%, and an acrylic content of at least 20% by weight based on the total weight of polymer.
2. A process as claimed in claim 1, wherein said particles are formed from a monomer mixture comprising at least one multi-ethylenically unsaturated monomer.
3. A process as claimed in claim 2, wherein said monomer is allyl methacrylate.
4. A process as claimed in claim 3, wherein allyl methacrylate is incorporated into said particles at a level of from 0.2% to 5%, preferably 0.3% to 2 % by weight, based on the total weight of said particles.
5. A composition for use in a process as claimed in any one of the preceding claims, comprising a crosslinking resin which is an aminoplast resin, multi-functional hydroxyalkyl amide or multi-functional N-alkylol amide, and an aqueous dispersion of acrylic polymer particles formed from a monomer mixture one component of which is allyl methacrylate, wherein said particles have a THF gel content as determined by the method defined hereinabove of 67% or more, preferably greater than 70%, and an acrylic content of at least 20% by weight based on the total weight of polymer.
6. Use of a composition, as claimed in claim 6, in or as a prepress sealer composition.

# **Patentansprüche**

1. Verfahren zur Herstellung von Spanplatten unter Hitze und Druck, umfassend die Bereitstellung eines Faservlieses, Behandlung der Oberfläche dieses Vlieses, Heißpressen des Vlieses bei erhöhter Temperatur und erhöhtem Druck zu einem festen Spanplattenprodukt und Trennung dieser Spanplatte von der Presse, dadurch gekennzeichnet, daß das Verfahren die Behandlung der Oberfläche dieses Vlieses mit einer Zusammensetzung umfaßt, welche ein vernetzendes Harz aufweist, das ein Aminoplastharz, multifunktionelles Hydroxyalkylamid oder multifunktionelles N-Alkylolamid und eine wäßrige Dispersion von Acrylpolymerpartikeln aufweist; wobei diese Partikel einen THF-Gelgehalt, wie bestimmt nach dem oben definierten Verfahren, von 67% oder mehr, vorzugsweise mehr als 70%, aufweisen und einen Acrylgehalt von wenigstens 20 Gew.-%, bezogen auf das Gesamtgewicht des Polymeren.
2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Partikel aus einem Monomergemisch gebildet sind, das wenigstens ein mehrfach-ethylenisch ungesättigtes Monomeres enthält.
3. Verfahren nach Anspruch 2, dadurch gekennzeichnet, daß das Monomere Allylmethacrylat ist.
4. Verfahren nach Anspruch 3, dadurch gekennzeichnet, daß Allylmethacrylat in diese Partikel in einer Menge von 0,2 Gew.-% bis 5 Gew.-%, vorzugsweise 0,3 Gew.-% bis 2 Gew.-%, bezogen auf das Gesamtgewicht der Partikel, eingebracht ist.
5. Zusammensetzung zur Verwendung in einem Verfahren gemäß irgendeinem der vorhergehenden Ansprüche, enthaltend ein vernetzendes Harz, das ein Aminoplastharz, multifunktionelles Hydroxyalkylamid oder multifunktionelles N-Alkylolamid ist und eine wäßrige Dispersion von Acrylpolymerpartikeln, die aus einem Monomergemisch gebildet sind, deren eine Komponente Allylmethacrylat ist, wobei diese Partikel einen THF-Gelgehalt, bestimmt nach der oben definierten Methode, von 67% oder mehr, vorzugsweise mehr als 70% haben und einen Acrylgehalt von wenigstens 20 Gew.-%, bezogen auf das Gesamtgewicht des Polymeren.
6. Verwendung einer Zusammensetzung nach Anspruch 5 in oder als Vorpressungsdichtmittel-Zusammensetzungen.

# **Revendications**

1. Procédé de fabrication de panneaux en une composition de bois, sous l'effet de la chaleur et de la pression, qui consiste à mettre à disposition un mat fibreux, à traiter la surface dudit mat, à comprimer à chaud ledit mat dans des conditions élevées de température et de pression pour le transformer en un produit de panneau ayant une composition solide, et à retirer de ladite presse ledit panneau en la composition, caractérisé en ce que ledit procédé comprend le fait de traiter la surface dudit mat avec une composition comprenant une résine réticulable, qui comprend une résine aminoplaste, un hydroxyalkylamide multifonctionnel ou un N-alkylolamide multifonctionnel, et une dispersion aqueuse de particules d'un polymère acrylique; où lesdites particules ont une teneur en gel de THF, telle que déterminée par la méthode définie ci-dessus, de 67 % ou plus, de préférence supérieure à 70 %, et une teneur en acrylique d'au moins 20 % en poids par rapport au poids total du polymère.
2. Procédé selon la revendication 1, dans lequel lesdites particules sont formées à partir d'un mélange de monomères comprenant au moins un monomère à insaturation multiéthylénique.
3. Procédé selon la revendication 2, dans lequel ledit monomère est le méthacrylate d'allyle.
4. Procédé selon la revendication 3, dans lequel le méthacrylate d'allyle est incorporé dans lesdites particules en une quantité de 0,2 à 5 %, de préférence de 0,3 à 2 % en poids par rapport au poids total desdites particules.
5. Composition destinée à être utilisée dans un procédé selon l'une quelconque des revendications précédentes, comprenant une résine réticulable qui est une résine aminoplaste, un hydroxyalkylamide multifonctionnel ou un N-alkylolamide multifonctionnel, et une dispersion aqueuse de particules d'un polymère acrylique formées à partir d'un mélange de monomères dont un constituant est le méthacrylate d'allyle, où lesdites particules ont une teneur en gel de THF, telle que déterminée par la méthode définie ci-dessus, de 67 % ou plus, de préférence supérieure à 70 %, et une teneur en acrylique d'au moins 20 % en poids par rapport au poids total du polymère.

6. Utilisation d'une composition selon la revendication 6, dans une composition d'étanchéité pour traitement préalable au pressage, ou en tant que composition de ce type.

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